



CRYSTALLISATION TECHNOLOGY

Process Technology

The objective of this paper is to describe the basics in crystallisation technology and additionally explain the interchangeability of fats and oils, including the compounding and processing of margarine, spreads and shortening.

Processing equipment

In principle two different crystallisation methods are used in the industry today: scraped surface heat exchanger (SSHE) plants and chilling drum plants. Gerstenberg Schröder manufacture high pressure scraped surface heat exchangers under the name Perfector and Kombinator.

Traditionally, margarine was produced in a chilling drum plant, like the Diacooler/Complector plant. Today this type of machinery is used limited and mainly for the production of puff pastry margarine and flakes.

The SSHE plant is by far the most flexible of the crystallisation technologies in terms of crystallisation of different types of fat products. The Perfector and Kombinator plant is capable of producing a large variety of fat products, including consumer and industrial margarine types, shortening, vanaspati/ghee, recombined butter and dairy blends.

Consumer margarine includes: all purpose table margarine, soft table, reduced and low fat spreads and speciality products. Speciality products can be margarine containing flavours, spices or functional ingredients. Industrial margarine includes bakery products such as puff pastry margarine, cake and cream margarine, frying margarine and bakery improvers.

The SSHE is the heart of the crystallisation process line in which the emulsion is chilled and hereby crystallised. The Perfector and the Kombinator exhibits a number of unique features such as different knives systems ensuring efficient scraping off of the crystallising emulsion. Pin rotor machines or intermediate crystallisers can be used in combination or individually. These kneading units are employed in order to yield mechanical work to the product and hereby ensure homogeneity, plasticity and spreadability. In addition, the kneading units give the product time to crystallise without any addition of cooling. When producing margarine of different fat blends, i.e. fat blends containing different raw materials, the flexibility is needed since the crystallisation characteristics of the blends might differ from one blend to another.



GS Perfector



GS Kombinator

Margarine production

Margarine, like butter, can be characterised as a water in oil emulsion, i.e. the water phase is dispersed as droplets in the continuous fat phase. In general, the production of margarine can be divided into the following parts:

- Preparation of the water phase and fat phase
- Emulsion preparation
- Pasteurisation
- Chilling, crystallisation and kneading
- Packing and remelting

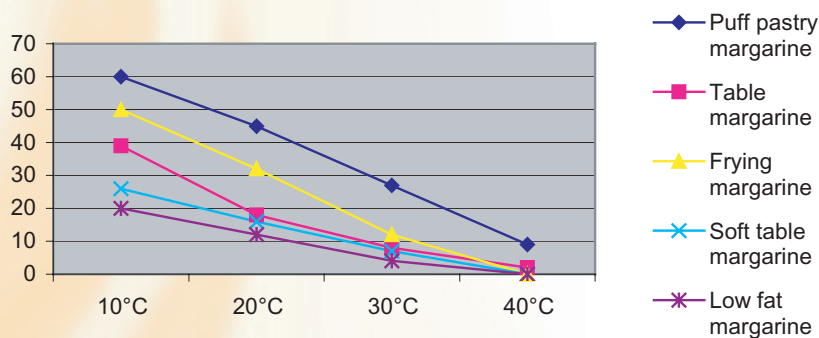
Minor ingredients such as emulsifiers, salt, preservatives, colour, flavour, antioxidants and vitamins are dispersed in the phases according to solubility. In addition, the extents to which additives may be used in food as well as the maximum dosage permitted often vary from country to country. Therefore, the legal authorities should always be consulted before using additives in food products.

The raw materials used in the emulsion preparation prior to processing of margarine can be divided into a fat phase and a water phase.

The fat phase

The major ingredients in the fat phase, the fat blend, consist normally of a blend of different fats and oils. When formulating a margarine blend, attention should be paid to the fat blend to achieve margarine with the desired characteristics and functionalities. The objective is to obtain a defined solid fat content (SFC) at various temperatures, typically ranging from 5°C to 40°C. The SFC profile will vary according to the type of product; tub margarine containing least solids at a given temperature and puff pastry highest amounts of solids.

The figure below shows the SFC profile of some margarine products.



Variations might occur within each product category, since margarine manufacturers have different product specifications.

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The ingredients used in the formulation of the fat blend can be derived from any animal, vegetable or marine oil source. The choice of fats will depend on legislation, economics, quality, functionality and on marketing constraints. The latter can limit the interchangeability of fats if e.g. 100% vegetable- or as now when low *trans* fatty acid margarines are in market demand. In addition, the availability of fats can be limited due to legislation, religious prohibitions, or trade barriers.

It is possible to divide ingredients into three main categories: liquid oils, semisolid fats, and hard stocks. A suitable blend can be made by combining one or more ingredients from each of the three groups. Later some recipe examples of margarine, spreads and fat products will be shown.

Liquid oils are considered to be completely interchangeable as components of margarine and shortening blends due to the modern refining processes. Fat modification processes, such as fractionation, hydrogenation, and interesterification enable a high degree of interchangeability among fats. The modification processes produce, individually or in combination, the full range of fatty intermediates used in the manufacture of all types of margarines and these processes enable fats to become almost completely interchangeable.

Liquid oils	Semisolid fats	Hard stocks
Unsaturated vegetable oils	Hydrogenated vegetable oils, MP: 32-34°C	Hydrogenated oils and fats, MP > 40°C
Palm olein	Hydrogenated marine oils, MP: 32-34°C	Interesterified blends, MP: > 40°C
Palm kernel	Palm	Beef tallow
Coconut	Lard	Palm stearin

A particular product specification can be met by a large number of alternative formulations. The producer attains the flexibility, in times of shortage or high cost, to interchange the best available and cheapest raw materials in an ever-changing market situation. The consumers are hereby certain to buy a product with continuously the same quality and price.

However, certain technical limitations exist when combining one or more ingredients from the 3 groups. The crystallisation habit, or polymorphism, of the used fat can set limits on the proportion of the particular fat used in the blend. In addition, the crystallisation rate of the fat blend is of great importance in the regard to the set up of the plant, in other words how much resting or holding time in the equipment the emulsion needs in order to be fully crystallised by the end of the processing line.

Crystallisation of fats

Fats tend to crystallise in various forms having different melting points. Each of these crystalline forms with their respective melting point are called polymorphs and the phenomenon is called polymorphism.

The triglycerides exhibit, with some exceptions, three basic crystalline forms designated alpha (α), beta prime (β'), and beta (β).

In general, transformations take place in the order: $\alpha \rightarrow \beta' \rightarrow \beta$

The transformations are irreversible except by melting and re-crystallisation. It is possible that the transformation from one polymorphic form to another takes place in the solid state without melting. This transformation will only take place in the direction of a more stable form, seeking the most compact crystal form and the lowest thermodynamic energy state possible.

The different polymorphs can be coexisting in the fat. These different forms show melting points depending on the cooling and heating history of the fat. Due to a so-called crystal memory, crystalline structure is preserved even though the fat is melted. This structure will affect crystallisation directly, especially when the rate of cooling is high. Polymorphic changes in the margarine may lead to a grainy structure.

When cooling the melt, α crystals are generally formed, but this form is never stable in triglycerides and transformation to β' is a reality. In most cases, β' crystals are relatively slowly transformed to the stable β form. The time of transformation from one crystal form to another depend on the composition of the triglycerides and the presence of diglycerides in the fat blend. However, some fats possess both β' and β forms, others only either stable β' form with no further transition or stable β form.

The literature states that the degree to which fats will exhibit either β or β' tendency depends on hydrogenation and depend to a lesser extent on blending factors. Also the palmitic acid content of the fat seems to be responsible for exhibiting a particular crystal habit. Fat containing relatively low amount of palmitic acid (C16:0), approximately 10%, seem to be β tending, where fats with at least twice the amount in general are β' .

Not only the amount of palmitic acid seems to determine the crystal habit, but also the distribution in the glycerol molecule. For instance, lard and tallow contain

approximately 24% and 25% palmitic acid respectively, but lard is β tending due to the high concentration of palmitic acid in the sn-2 position of the glycerol molecule whereas tallow is β' tending because of sn-1,3 positioning.

The table below shows crystal habits of selected fats.

β' tending fats	β tending fats
Butter oil/milk fat	Canola
Coconut	Cocoa butter
Cotton seed	Corn
Modified Lard	Lard
Palm	Soyabean
Tallow	Sunflower

Source: Wiedermann

Apart from a few types of shortening the β' crystals are the most desirable. They are relatively small and can incorporate a larger amount of liquid oil in the crystal network. β' crystals result in a glossy surface and a smooth texture.

The transition from the α -form to the β' -form takes place in the crystallisation equipment. All fats tend to crystallise in the β' -form when crystallisation takes place in a SSHE plant. Fats that are not stable in the β' -form due to its triglyceride composition will during storage transform into the β -form. The rate of this transition is increased by temperature fluctuations.

Minor ingredients in the fat phase

Apart from the fat blend, the fat phase consists of minor ingredients such as emulsifier, lecithin, flavour, colour, and antioxidants. These minor ingredients are dissolved in the fat phase before emulsification.

Emulsifiers are surface-active compounds and are used to reduce the interfacial tension between the water and the fat phase. The emulsifier stabilises the liquid emulsion before crystallisation to secure a homogeneous product and to achieve a finely dispersed and stable water distribution in order to improve the microbiological keeping properties in the margarine. Furthermore, addition of emulsifier ensures that the emulsion is heat stable during baking.

The most commonly used emulsifiers in margarine are monoglycerides or mixtures of mono- and diglycerides. The best water binding effect in margarine is achieved by saturated monoglycerides. If a blend of mono- and diglycerides is used, it is important to remember that it is only the monoglycerides that have the emulsifying effect and the total amount added should therefore be larger than if a distilled monoglyceride was used.

Soya lecithin can be used with the emulsifier to improve the effects of the emulsifier. To achieve the maximum effect of the emulsifier-lecithin system, the emulsifier is heated with the lecithin and liquid oil before the mixture is dissolved in the melted fat blend. Additionally, lecithin reduces spattering in frying margarine, and it prevents burning of sediment compounds (lactose and casein) since lecithin incorporate these compounds. Lecithin is also a good antioxidant.

Fat soluble flavour and colour, e.g. butter flavour and β -carotene respectively, are added to achieve a product that tastes and looks like butter. In addition, β -carotene has pro-vitamin A activity.

Antioxidants can be added in order to improve the shelf life of the product, since these substances can delay the onset or slow the rate of oxidation. In margarine production the following antioxidants can be added to the water or fat phase according to solubility: natural tocopherols, BHA (Butylated hydroxyanisole), BHT (butylated hydroxytoluene), Ascorbic acid, and Ascorbic palmitate.

The aqueous phase

The water phase differs depending on the type of margarine, thus it has a certain influence on the finished margarine. It consists mainly of water in which the minor ingredients such as salt, preservative, milk protein and conceivably sugar are dissolved. Water-soluble flavor and color can also be added, but are primarily used in low fat spreads.

Salt is added primarily to improve taste, but also to prevent growth of microorganisms. In frying margarine, salt helps to prevent spattering. The amount added varies from 0.2% to 2.5%.

Citric acid is used to lower pH which means it acts as a preservative but additionally concerning puff pastry margarine, the low pH will help give the finished pastry a better lift.

Other preservatives often used in margarine are benzoates and sorbates. They are most active at pH around 4.5 which makes the advantage of adding them doubtful due to the fact that pH in most margarine emulsions is often close to neutral.

Milk proteins such as skim milk powder, whey powder, cream powder or sweet butter milk powder have an O/W emulsifying effect, which means that these ingredients work against the margarine W/O emulsifier system, and thus destabilises the margarine emulsion. However this enhances the flavor release and milk protein is added to the emulsion when producing table margarine and reduced calorie spreads to influence the taste. Milk proteins give additionally the desired browning effect in margarine used for frying.

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In 80% margarine, preservatives are not necessary as long as the product is properly crystallised and packed. If the water droplets are finely distributed and their size are between 1 to 5 microns, microorganisms cannot grow.

There are a large variety of stabilisers or hydrocolloids on the market. When the manufacturer chose the stabiliser, the water binding capacity and organoleptic properties has to be considered. Typically alginate, pectin, carrageenan, starch and mixtures are used.

Low fat spreads

During the last decades, low fat spreads have become very popular due to an increasing awareness in the population concerning the degree of fat intake.

Back in the late 60's, the first margarines with a fat content reduced to approximately 40% were produced. Thus, these products contained only 50% of the normal fat content and consisted of only fat, water, salt, emulsifier, flavor, vitamins and preservatives. These products showed poor melting and flavor release properties due to very tight water-in-oil emulsions. This tight emulsion was required in order to have the needed stability.

In the early 80's, 40% fat products containing very high amount of milk solids were marketed in the US. These products exhibited, however, very poor microbiological stability. Consequently, these products did not have the 4 to 6 months shelf life like other spreads and were therefore withdrawn from the market. Today, higher quality products containing lower fat, usually milk proteins and stabiliser systems are being produced with great success in many countries.

Some recipe suggestions for low fat spreads, i.e. products containing 40% fat of the total emulsion, are shown below.

Fats and oils	1	2	3
Hydrogenated vegetable oil MP 41/42°C	25 parts	20 parts	-
Hydrogenated vegetable oil MP 35/36°C	-	20 parts	-
Interesterified blend MP 41/42°C	-	-	30 parts
Liquid oil	75 parts	60 parts	70 parts

Liquid oil: soybean oil, cottonseed oil, palm olein, canola (rapeseed) oil etc.

Low fat emulsions have been found to be sensitive to in-line pressure and cooling rate. If too extensive chilling takes place early in the process, the shearing forces might become too large and the emulsion might break. Therefore, low fat products should be produced by high-liquid, low SFC profile blends to minimise this problem.

Emulsion preparation

Initially, the emulsion is prepared. For low fat spreads as for other types of margarine, the various fats of the specific blend are melted. Usually, the high melting fats or fat blends are added first followed by the lower melting fats and the liquid oil. To complete the preparation of the fat phase, the emulsifier and other oil-soluble minor ingredients are added to the fat blend. When all the ingredients for the fat phase have been properly mixed, the water phase is added and the emulsion is created under intensive but controlled mixing. It is important when producing low fat spreads that the aqueous phase and the oil have similar temperature and are combined slowly when forming the emulsion. Additionally, it is very important that the emulsion is properly agitated to ensure homogeneity. However, care should be taken not to incorporate air during emulsification. Prior to entering the crystallisation equipment, the emulsion is pasteurised either in a plate heat exchanger (PHE) or in a low pressure SSHE, the Consistator®, or in a high pressure SSHE, the Kombinator or Perfector.

Depending on the composition of the emulsion and especially the aqueous phase, low fat emulsions can be more or less stable. Stable, viscous emulsions are easy to crystallise. Relatively low cooling temperature can be applied and the product can be produced like normal table margarine.

Leaving the last chilling section the product is ready for filling. The filling temperature is normally higher for low fat products than the corresponding 80% fat products since the emulsion is more viscous. If the filling temperature is too low, the final product may become crumbly with water leakage as it is packaged.



GS Pin rotor machine

Crystallised fat products

Turning to the high fat products, some general recipe suggestions for various margarine types are listed below.

Fats and oils	Table	Soft table	Puff pastry
Palm stearin	-	-	62 parts
Interesterified blend MP 41/43°C	40 parts	20 parts	25 parts
RBD palm oil	20 parts	20 parts	13 parts
Liquid oil	40 parts	60 parts	-

Liquid oil: soybean oil, cottonseed oil, palm olein, canola (rapeseed) oil etc.

The recipe for puff pastry margarine is compounded to be used at approximately 25°C. Puff pastry margarine has the features of exhibiting a very good plasticity without being oily. The plasticity of the finished margarine depends on the crystallisation process, on the composition of the fat blend and on the processing conditions. The puff pastry recipe contains a relatively high amount of palm stearin, since puff pastry margarine containing palm oil exhibits excellent plasticity.

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However, in some areas of the world, palm oil as a raw material for margarine is not an option, and other raw materials have to be used. By substituting palm stearin with another hard stock which could be hydrogenated vegetable oil or an interesterified hard stock, a suitable puff pastry margarine fat blend could be made.

In regard to fat products not containing an aqueous phase, recipe suggestions for shortening and vanaspati will be described below.

Ingredients	Shortening	Vanaspati
Palm stearin	10 parts	-
RBD palm oil	40 parts	90 parts
Intesterified blend MP 41/43°C	30 parts	-
Liquid oil	20 parts	10 parts

The shortening is an all purpose shortening with a relatively high melting point. Shortening is an industrial product, exhibiting a plastic and smooth texture. When producing shortening, N₂ can be added in order to give the product a nice white appearance and make it easier to handle.

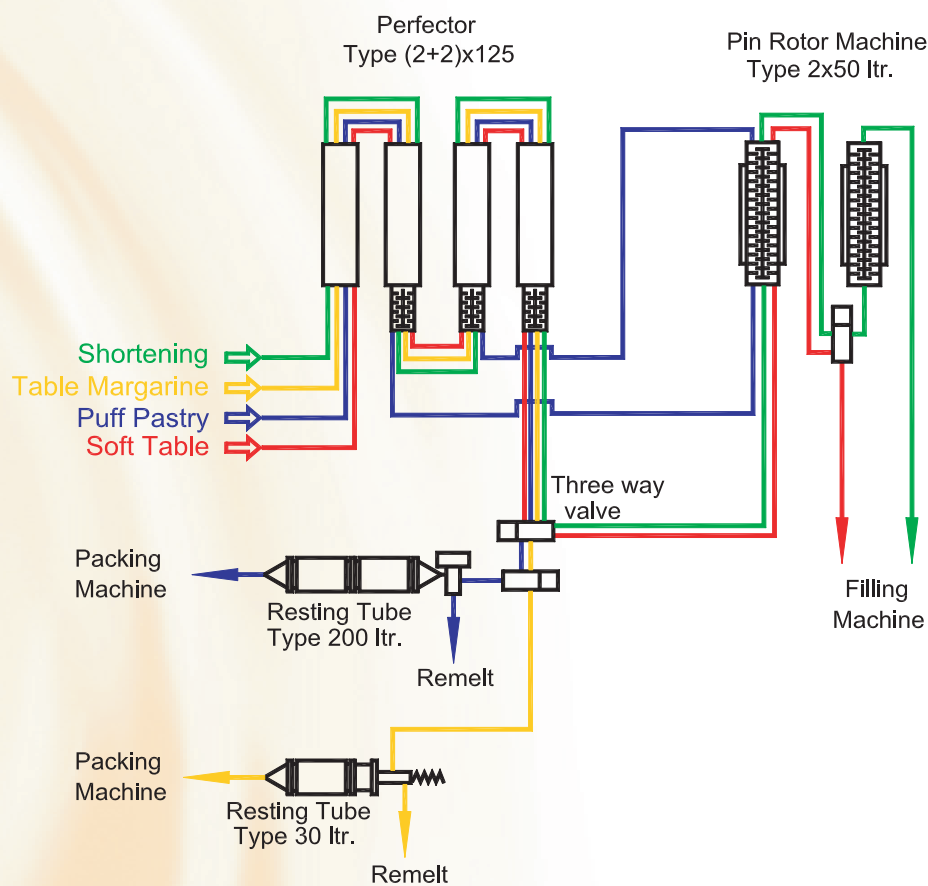
Where shortening is only used for cooking and baking, vanaspati or ghee is used as a multipurpose fat in the households in Africa, Middle East, Pakistan, India, and Bangladesh. Traditionally, this product called ghee consisted of 100% animal fat, originating mainly from cow and buffalo. However, due to the positive health claims of vegetable fats and lower price of the raw materials compared to the traditional ingredients, a large amount of the product sold today contains 100% vegetable fat. The traditional ghee has a granular appearance, thus the vanaspati produced today by vegetable fats has to exhibit the same texture. The recipe for vanaspati shown above is a 100% vegetable product. The ratio of RBD palm oil to liquid oil can vary, and RBD palm oil can be substituted by other semisolid fats, such as partially hydrogenated vegetable oil.

When all the ingredients are mixed and properly emulsified as previously described, the emulsion is transferred from the mixing tank to the buffer or run tank. The emulsion is then pumped through the crystallisation equipment, where sub cooling and later crystallisation takes place. The Perfector plant shown on the flow diagram consists of 4 chilling tubes with 2 independent cooling systems and is mounted with 3 intermediate crystallisers.

The plant has 2 separate pin rotor machines. In case of shortening which is filled into containers as semi-liquid, the product flow would go through both pin rotor machines. In case of soft margarine filled in plastic tubs, the product flow would go through one pin rotor machine just before cup filler. In case of margarines wrapped in paper, for table margarine or puff pastry margarine, one or both pin rotor

machines, depending on the oil blend and the desired plasticity of the product, would be placed in between the chilling tubes.

It can be noted that only minor changes is needed in order to produce a variety of products. The plant described here can produce any kind of margarine and shortening, as well as all kinds of oils and fats can be used as raw materials. The only change and adjustment to be done when switching from one product to another is rearranging the pipe connections and off course also the processing conditions. By the right design of the cooling equipment with crystallisers, resting tubes etc. and control of the temperature parameters, it is possible to manufacture a high quality product with the desired texture, no matter which raw materials are used.



***Trans* fatty acids**

Traditionally, the margarine fat blend consists of partially hydrogenated vegetable oil in certain ratio to the liquid oil, as described earlier. In general, hydrogenated fats crystallise relatively faster than unhydrogenated fats and provide the finished margarine with the required characteristics. But during the hydrogenation process various *trans* fatty acids are formed and these isomers are nutritionally undesirable. Scientific research indicates a correlation between a certain intake of these isomers and the risk of cardiovascular heart diseases.

The main direct sources of *trans* fatty acids in the human diet is margarine and crystallised fat products. Indirectly sources cover products such as baked products, fried foods etc.

Partially hydrogenated oils may contain up to 55% *trans* fatty acids, depending on the source of oil, the refining and hydrogenation process. The higher degree of unsaturation, the higher amount of *trans* fatty acids will be present after the partial hydrogenation process all other things being equal.

Animal fat contains naturally up to 5% *trans* fatty acids, which occur due to the fermentation processes done by microorganisms in the rumen of ruminants. Deodorised oils may contain up to 3% *trans* fatty acid due to the high temperature this process is performed under.

It is possible to produce non *trans* vegetable oil intermediates by the classical modification processes, such as fractionation, hydrogenation and interesterification. These fats will still fall into the three main groups of liquid, semisolid, and hard stock fats.

The increase in the melting point done by hydrogenation process is due to bond changes, either due to an increase in saturation or change in the configuration from *cis* to *trans*. However, certain potential disadvantages arise with *trans* free formulations. Blends consisting of low *trans* fats, i.e. *cis* blends, have shown a decrease in oxidative stability when compared to similar blends containing *trans* fatty acids. Additionally, since *trans* fatty acids have a higher melting point than the corresponding *cis* fatty acids, the amount of saturated fatty acids in the blend have to be increased in order to achieve blends with the desired melting point.

The global trend of lowering the amount of *trans* fatty acids in food products continues at great speed and the food industry worldwide is decreasing the use of partially hydrogenated fats. The driving force has been the multiple scientific research published on the issue, and the legislative initiatives, e.g. the ban of *trans* fats in Denmark by 2004 and the FDA labelling rule effective by January 2006. The majority of the larger players in the fats and oils industry (e.g. ADM, Frito-Lay, McDonalds, Nestlé) have already shifted to low *trans* products, however certain parts of the world are not yet focused on the problem.

Quality of margarine

In the case of food products, quality falls into two groups: apparent and hidden quality. Apparent quality is what the customer perceives and hidden quality is values that cannot be judged sensorically. Nutritive value and no presence of dangerous microbiological or chemical contaminations are examples of hidden quality values. The manufacturer has to guarantee these values which most often are specified by legislation.

The hidden quality is a specific term compared to apparent quality since it covers measurable values and standardised analytical methods are available.

The apparent quality varies among the margarine manufacturers and there is not much literature available dealing with this issue most likely because of competition. However, margarine can be evaluated according to its flavour, texture, mouthfeel and neatness of packaging. In addition surface appearance, oil/water separation, grainy texture, sticky mouthfeel, meltdown of the margarine and flavor release has to be rated when the margarine is evaluated.

The various types of margarine are evaluated in regard to their use.

The spreadability of table, tub and low fat margarine and spreads is a very important quality parameter. The homogeneity, consistency, gloss, sandiness, loss of water can hereby be determined.

The stability of table margarine and spreads can be described by the spreadability test. The test involves spreading of the sample with a knife on cardboard, and the following rating system where values from 5-1 can be used. The value 5 describes a highly stable and very smooth sample, the value 3 describes a sample that separates when worked, 1 describes a sample that separates during processing where e.g. free water is visible.

When producing low and reduced fat spreads, the eating properties should be similar to those of full fat soft tub margarines in terms of mouth-feel and flavor release. In addition, the products should be spreadable directly from the refrigerator.

Mouth-feel and flavor release can individually be rated as follows: fast, average and slow.

Margarine which apart from being used for spreading on bread additionally is used for cooking should further be evaluated for functionality. Frying margarine should be evaluated in regard to spattering and surface burning of the pan. In regard to industrial margarine and shortening, the appearance is less important, the functionalities being the significant properties and consequently the evaluation should be concentrated on these matters.

Cake and cream margarines are evaluated in regard to the creaming performance. Margarine is whipped with sugar according to a standardised procedure and by weighing a known volume the ability to incorporate air is then calculated. Flavor is important for all types of margarines, but in the case of margarine and shortening used for baking, the flavor has to exhibit certain heat stability in order not to disappear completely during baking. In the case of puff pastry margarine, the best way to evaluate this product is by test baking. But several other objective and subjective methods are available.



When producing puff pastry, a basic dough is rolled out and the roll-in margarine, the puff pastry margarine, is placed on the dough in one flat piece and following completely covered with the dough. Several folding and rolling procedures follows in order to produce multiple alternate dough/margarine layers. These layers result in products with flaky structure. The flakiness depends on the formation of thin films of gluten which trap water vapor and carbon dioxide from fermentation. These laminates can form a three-dimensional structure and for this not to occur, the laminates must be separated by continuous sheets of fat. Thus fat works as a barrier between the basic dough layers and prevents them from joining while they are being reduced in thickness during the folding and rolling procedure. To function as a barrier the fat should not be absorbed by the dough layers, and it must remain as a continuous fat film throughout the rolling process.

The main demands to puff pastry margarine are therefore plasticity and firmness, since soft and oily margarines tend to be absorbed by the dough and hard and brittle margarines are difficult to spread between the two dough layers during the rolling procedure. In both cases the baking performances may be affected negatively. Puff pastry margarine is by the professional baker evaluated by a subjective method called finger evaluation of plasticity, which include a thumb test and a kneading test. The thumb test is performed by pressing the thumb several times in adjacent areas in a slice of margarine. This gives an indication if the margarine is firm and homogeneous without lumps.

The test of kneading is a method that determines the body of the margarine and is accomplished by working or moulding a piece of margarine. A slice is kneaded by hand for a couple of minutes. If the sample retains toughness it indicates that the sample will be able to withstand the rolling procedure. The bending test is another subjective method showing whether a slice of margarine can be bent without cracking. If the margarine can be bent, the margarine is evaluated to be plastic.

Good baking performance is not always in accordance with subjective evaluation, since the margarine that was initially evaluated as being of poor quality can perform very well in baking. However, it can be difficult to persuade the baker to use the margarine if the subjective evaluation does not meet the standard of the specific baker. Consequently, the evaluation of plasticity can be very important in regard to the description of the quality. The baking test is performed in order to evaluate the abilities of the puff pastry margarine as a roll-in fat. The height of the pastry and the uniformity of the lamination are evaluated.

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It is very important in the case of evaluating margarine to define the quality parameters, since these vary from country to country, sometimes even from region to region. Also, most of the above-mentioned evaluation principles are based on subjective procedures but objective methods are available like the creaming performance test described. In addition, Texture Analyzer equipment, like the TA TXplus, can be used to objectively describe the plasticity, stickiness, and homogeneity of the margarine. Standard methods are used in the industry today but complementarily to the „old-fashioned“ subjective evaluation principles.



Please contact GS for further information.

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